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# Structural Refinement of the individual grains in a polycrystal

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Today structural refinements are based on either single crystal or powder diffraction data. In many cases neither method is applicable. Specifically, it may be difficult to grow single crystals or the grains need to be studied within their environment. Furthermore, as it is possible to solve and refine much more complex structures from single crystal data than from powder data, it is essentially always of interest to be able to obtain the former sort of data.

We therefore propose to treat the individual grains in the sample (powder, pellet or component) as a collection of single crystals. An indexing and integration program, GRAINDEX, is presented which can sort diffraction spots with respect to grain for hundreds of grains simultaneously (Lauridsen *et al.* 2001). The only limitations are the grain size and the amount of overlap between diffraction spots.

The method is implemented at beamline ID11 at ESRF. Used with hard x-rays (50-100 keV) the extinction and absorption are negligible, while grains of order 300 nm are visible. The method has been initially validated by a study of $Al_2O_3$ (corundum). An annealed pellet with an average grain size of 20 μm was illuminated with a 50 keV 250 x 250 $\mu m^2$ beam. Rotating the sample around one axis with a step-size of 0.1 degree a total of 80.000 spots were detected. Based on an intensity cut 57 of the most intense grains were refined with a typical $R_{sym}$ of <10% after appropriate scaling and filtering. Structural refinements carried out on the thereby obtained integrated peak intensities are essentially identical to that of single crystal data. Studies of more complicated structures are currently underway.

Compared to powder diffraction the method has advantages, in addition to the complexity of the structures which may be treated:

- Instead of average information, distributions are derived.
- Minority phases occupying volume fractions of $10^{-8}$ or less can be detected.
- Refining individual grains is a natural extension of the 3DXRD concept (Lauridsen *et al.* 2001, Poulsen *et al.* 2001). From 3DXRD the position, volume, crystallographic orientation and strain state of a set of grains are determined simultaneously. Likewise 3D maps of grain boundaries can be derived. Hence,

cross-correlations to other types of structural information can be performed.

## References

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